



IRmadilloTM
Process Analyser



How to calibrate the IRmadillo spectrometer

Introduction

The IRmadillo is a “static optics” FTIR spectrometer. It has been specifically designed to operate in industrial environments, giving the quality of information typically only found in laboratory environments – right on the manufacturing floor or process plant. Spectroscopy is an incredibly powerful tool for process analysis, but it does require calibration to become a universal chemical-concentration analyser.

Keit can work with you to provide this calibration service, and this handout explains the “dos and don’ts” of calibration, as well as what is required at each step.

Chemometrics vs Peak Fitting

Traditional spectroscopy measurements would simply look at the height of a peak in a spectrum that was caused by the chemical of interest and measure its height or area with regards to concentration and create a simple calibration curve of x vs y. This is called “univariate analysis”. It’s a very simple technique and for simple mixtures can be very powerful. But, it relies on clearly defined spectral features, linear behaviours and quite high concentrations of chemicals.

Modern spectroscopic measurements use a type of statistical analysis called “multivariate analysis” (also called “chemometrics”). This involves analysing the entire spectrum at once, and pulling out the corresponding “spectra within spectra” that correspond to the chemicals of interest. This typically uses either Partial Least Squares (PLS) or Support Vector Machine Regression (SVR) calibrations depending on whether the calibration curve is linear or not.

Three Golden Rules of Calibration

Calibrating any measurement technique based on spectroscopy should follow three simple rules:

1. Calibrations need variation in chemical concentration

It is important to make sure that the chemicals of interest can vary throughout the entire concentration range. For example, if the chemical of interest can change between 5 and 15 % in a typical process then a calibration that was only built between 8 – 10 % would need to extrapolate outside of its calibration space, and is likely to give poor results.

2. Calibrations need every chemical of interest to change concentration independently

If the concentrations of two chemicals are linked (either correlated or anti-correlated) then the calibration cannot separate out their spectra. It’s important that they are allowed to change concentration independently during calibration – using laboratory prepared samples if necessary.

3. Calibrations are only as good as the reference data used to build them

If the reference data from off-line laboratories (such as HPLC/GC-MS or chemicals assays such as titrations) have errors, then these will be transferred into the calibration. So if poor results are fed into the calibration, then poor results will come out of it. It may be necessary to look into third-party laboratory services for the calibration phase to ensure that reliable, robust and trustworthy analysis is being used to build the calibration.

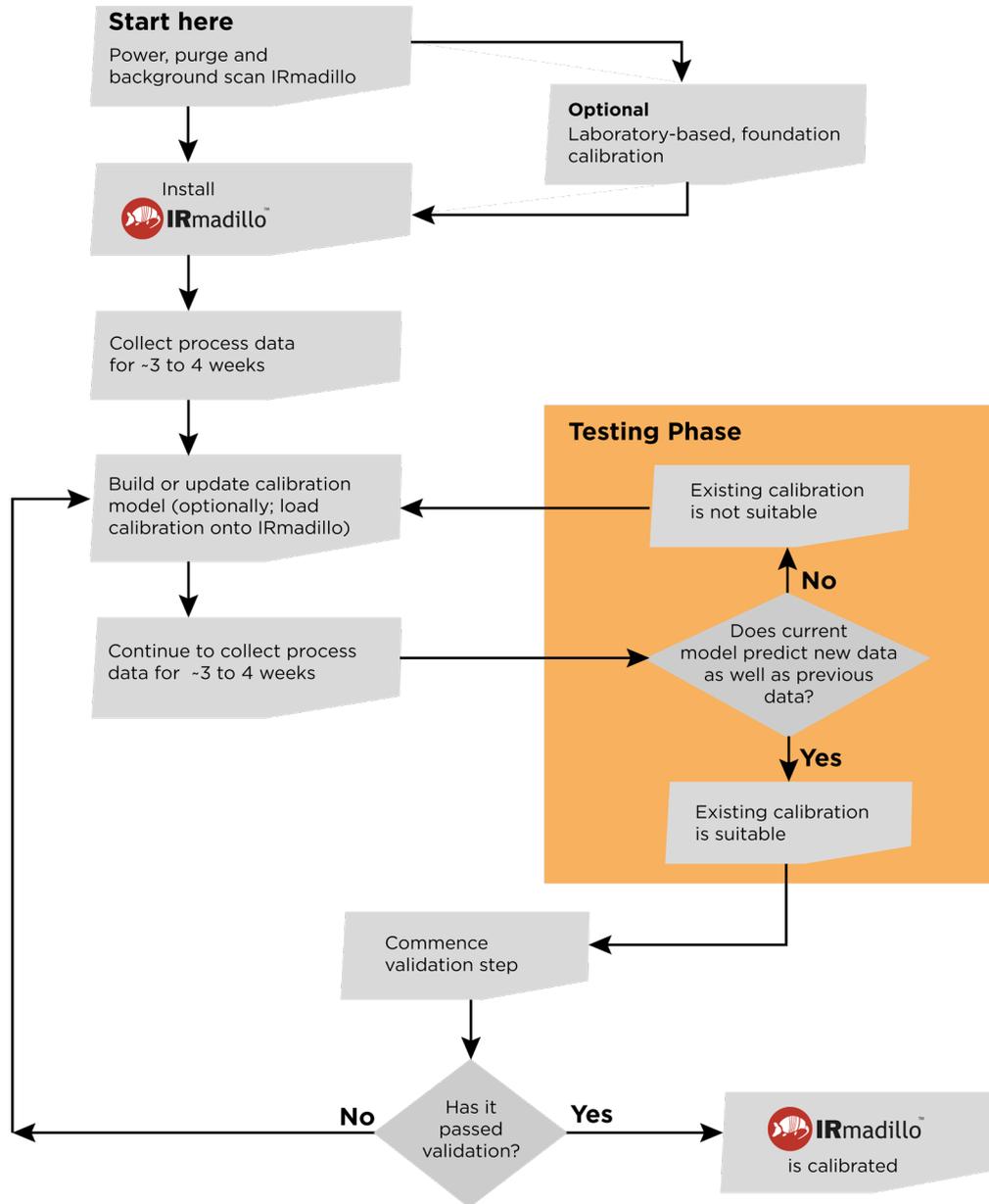
Keep in mind the IRmadillo can also be calibrated for qualitative measurements

Sometimes it is not possible to build a calibration for quantitative measurements. This might be because it’s not possible to actually measure the chemical of interest using off-line measurements, either because it cannot be removed from the analyte, it is unstable or something else.

In that case, it may be practical to create a qualitative measurement. This could be calibrated to when the plant is running well and called a “high performing state”, and an alternative calibration built for the “low performing state”. By using “classification models” such as Partial Least Squares Discriminate Analysis (PLS-DA) it is possible to see when the “high performing state” starts to drift towards “low performing state” and take action to correct it.



Calibration Process



How long does it take?

It depends on the variation that's present in your process. But if the golden rules are followed, and with the right planning (such as laboratory foundation pre-calibration when required), it should be possible to deliver an operational and calibrated IRmadillo within a matter of weeks.

Who do I contact?

Get in touch with us to find out more.

- +44 (0)1235 431260
- enquiries@keit.co.uk
- www.keit.co.uk